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PRELIMINARY RESULTS FROM SCREENING TESTS OF COMMERCIAL CATALYSTS WITH POTENTIAL USE IN GAS TURBINE COMBUSTORS
PART I. FURNACE STUDIES OF CATALYST ACTIVITY

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ABSTRACT

Thirty commercially produced monolith and pellet catalysts were tested as part of a screening process to select catalysts suitable for use in a gas turbine combustor. The catalysts were contained in a 1.8 centimeter diameter quartz tube and heated to temperatures varying between 300 and 1200 K while a mixture of propane and air passed through the bed at space velocities of 44 000 to 70 000 hour⁻¹. The amount of propane oxidized was measured as a function of catalyst temperature. Of the samples tested, the most effective catalysts proved to be noble metal catalysts on monolith substrates.

PRELIMINARY RESULTS FROM SCREENING TESTS OF

COMMERCIAL CATALYSTS WITH POTENTIAL USE

IN GAS TURBINE COMBUSTORS

PART I. FURNACE STUDIES OF CATALYST ACTIVITY

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SUMMARY

The tests described here were part of a process to select catalysts which would be suitable for use in a gas turbine catalytic combustor. Thirty monolith and pellet catalysts manufactured by seven companies were tested in a tube furnace to determine the effect of catalyst temperature on the oxidation of propane. Noble-metal, base-metal, and rare-earth-oxide catalysts were included. They were contained in a 13 cubic centimeter volume within a 1.8 centimeter diameter quartz tube. Space velocities (volumetric flow rate at standard conditions divided by the overall catalyst volume) were maintained between 44 000 and 70 000 hour⁻¹, pressure was 2×10^5 newtons per square meter and temperatures were varied between 300 and 1200 K.

For these test conditions and this test configuration, thermal oxidation of propane occurred for temperatures greater than 1050 K. The propane was completely oxidized at 1175 K.

The lowest temperature at which catalytic oxidation took place varied between 325 and 750 K depending on the catalyst and manufacturer. Noble metal catalysts from Engelhard and Grace oxidized over 90 percent of the initial propane concentration at a temperature of 650 K.

A change in space velocity from 44 000 to 61 000 hour⁻¹ with constant catalyst volume was shown to decrease the propane oxidation by only about 10 percent, while a doubling of the catalyst volume with constant mixture flow rate had no apparent effect on the amount oxidized.

The catalysts tested were fresh samples. Further testing will be required to determine the effect of aging at temperatures of 1400 to 1600 K on the catalytic activity before selection of the most suitable catalysts for catalytic combustion can be made.

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INTRODUCTION

The test results presented in this paper were obtained as one element in a program to evolve catalytic-combustor technology for an automotive gas turbine engine. In this preliminary screening study, a number of commercially produced catalysts were tested to establish the effect of catalyst temperature on the propane oxidation efficiency.

The need for gas turbine combustors with good combustion efficiency but producing minimal concentrations of nitrogen oxides has led to the consideration of catalytic (heterogeneous) combustion as a possible alternative to thermal (homogeneous) combustion. The concentration of nitrogen oxides (NO_X) produced by the oxidation of nitrogen in the air during combustion is shown in figure 1 to be nearly exponentially dependent on flame temperature. The curve of figure 1 is an analytical prediction which assumes that a homogeneous fuel-air mixture burns in a well-stirred reactor. The model is described in reference 1. Experiments in which fuel was prevaporized and premixed with air before combustion have confirmed this predicted curve for temperatures greater than 1800 K (ref. 2).

Current gas turbine combustors produce on the order of 100 to 500 ppm by volume of nitrogen oxides because they operate with a nonhomogeneous fuel-air mixture and with high peak flame temperatures. In contrast, a premixed, prevaporized combustor has been tested at steady-state conditions (ref. 3) and shown to produce minimum NO_X emissions of about 0.3 g NO₂/kg fuel (6 ppm by volume) with a measured primary zone flame temperature of about 1800 K. Figure 1 shows that an additional order-of-magnitude reduction might be achieved if combustion temperatures could be reduced to around 1600 K. However, because the lean flammability limit for hydrocarbon fuels occurs at a flame temperature of around 1800 K (ref. 2), combustion at lower flame temperatures is not possible without some means of combustion augmentation.

By increasing the rate of reaction, a catalyst permits combustion to take place at temperatures well below the flammability limit; therefore, a catalytic combustor has the potential for good efficiency with extremely low nitrogen oxides emissions.

The catalytic combustor will consist of two major components: a fuel-air preparation system to supply a homogeneous fuel-air mixture, and a catalytic reactor capable of fully reacting the fuel and air at low temperatures. No systematic studies of fuel-air preparation to achieve uniform mixtures have been published to date, but a number of catalytic reactor feasibility tests have been performed (refs. 4 to 7). The results of these tests showed that good efficiency was possible with NO_X concentrations near 1 ppm or less, but only over narrow ranges of operating conditions.

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NASA-Lewis Research Center has been helping to evolve the technology required for the catalytic combustor as part of an automotive gas turbine engine program supported by the Energy Research and Development Administration. Our program includes an evaluation of fuel nozzle configurations to establish a fully vaporized, homogeneous fuel-air mixture at the reactor inlet. Reference 8 is a preliminary report of this study. Reactor studies involve the screening of commercial catalysts to determine their suitability for use as hydrocarbon oxidation catalysts at gas turbine operating conditions.

The first part of the screening study involved the evaluation of both pellet and monolith catalysts in small quantities in a tube furnace to establish the most active ones. Although test data has been reported for pure metal and metal oxide catalysts (refs. 9 and 10), no published data exists on the hydrocarbon-oxidation capabilities of commercially available catalysts. Preliminary results of the furnace-screening tests are reported here. Catalysts selected from this study were then further tested in a combustion test rig at combustor operating conditions (ref. 11). In addition to this in-house program, Lewis also has a contract with Engelhard Industries (NASA Contract No. NAS3-19416) to demonstrate the durability of catalysts capable of operating at gas turbine combustor conditions.

In the tests reported here, catalyst samples were heated in a tube furnace to temperatures ranging from 300 to 1200 K. Approximately 500 ppm by volume propane in air was the feed gas to the heated catalyst at space velocities (volumetric flow rate at standard conditions divided by the overall catalyst volume) of 44 000 to 70 000 hour⁻¹. The corresponding inlet velocities were on the order of 1 meter per second. The pressure was 2×10^5 newtons per square meter and catalyst volumes were 13 cubic centimeters. Exhaust concentrations of unburned hydrocarbons and carbon monoxide were measured. Although catalysts in the form of a monolith are likely to be more suited than pellets for gas turbine catalytic combustors, both forms were tested in order not to overlook any pellet forms which might prove exceptionally good. None of the catalysts were aged before testing.

APPARATUS AND PROCEDURE

The test apparatus consisted primarily of a tube furnace for heating a catalyst. A dilute mixture of propane in air was passed through the catalyst to determine the efficiency of oxidation as a function of catalyst temperature. Figure 2(a) shows the test equipment schematically.

The fuel-air mixture was supplied from commercially prepared bottles normally used as calibration gases for hydrocarbon analyzers. Concentration was

usually about 500 ppm propane in air. Several test runs were also made in which a concentration of 280 ppm was used, but no effect of fuel concentration on the fraction oxidized-versus-bed temperature curve was observed. The ideal temperature rise for 500 ppm propane burned in air is about 30 K; therefore, the temperature rise across the catalyst resulting from fuel oxidation should be small compared with the bed temperature determined by the furnace.

The catalyst was contained in a section of 1.8-centimeter inside diameter quartz tubing, shown in figure 2(b). The tubing was hung vertically in a Marshall tubular furnace so that the catalyst was near the center of the furnace. The tubing was supported by a stainless-steel framework as shown in figure 2(c).

When monolith samples were tested, the sample was wrapped with quartz wool to prevent leakage of fuel-air mixture around the outside of the bed. Clearance between the catalyst and quartz tubing was small, however, and several tests made with beds wrapped in this way and unwrapped showed no effect on the results. Upstream of the catalyst were eight units of 2.5 centimeters long inert cordierite monolith substrate to heat the inlet gas mixture to near the bed temperature.

Figure 3 shows typical samples of some of the pellets and monoliths tested. Table I gives a description of the pellet catalysts and table II gives information on the monoliths tested.

Various sizes and shapes of pellets were tested. Total volume of the pellet bed, including a thermocouple located in the middle of the bed, was 13 cubic centimeters.

Monolith test pieces were 1.8 centimeters in diameter and 5 centimeters long. They were drilled along the centerline to a depth of 2.5 centimeters so that a thermocouple could be inserted from downstream. This thermocouple touched the catalyst but was not cemented to it; therefore, the recorded bed temperature is dependent on both the bed surface temperature and the gas temperature.

A second thermocouple was located at the downstream end of both pellet and monolith beds to determine if any large temperature gradients existed in the bed.

Exhaust gases from the catalyst passed through 10 meters of 0.6 centimeter diameter stainless steel tubing to the gas analysis equipment. Typical traverse times were less than 5 seconds. The gases were analyzed to determine the concentrations of unburned hydrocarbons using a Beckman Model 402 flame ionization detector; carbon monoxide concentration was measured with a Beckman Model 315B nondispersive infrared analyzer. The hydrocarbon analyzer was calibrated with the same propane-air mixture that was used for testing. A bypass line connected the fuel-air supply with the analyzer to permit calibration checks while the catalyst was hot.

Experimental data were taken while the furnace was cooling. Cooling from about 1200 K to near room temperature took about $3\frac{1}{2}$ hours. During this time data were taken at desired temperatures by opening the fuel-air mixture flow valve and allowing about 1 minute for the analyzers to reach steady-state values. While the mixture was flowing the cooling rate of the catalyst bed was as high as 1 K per second because the inlet stream was at a lower temperature than the bed. The fuel-air mixture flow rate, the temperatures, and the emissions were recorded in less than 2 seconds with an automatic data acquisition system. Added to the 5-second transit time from the catalyst to the analyzers, the delay could result in as much as 7 K difference between the recorded bed temperature and the bed temperature which corresponded with the recorded emissions. This difference is negligible.

RESULTS AND DISCUSSION

Propane Oxidation

The expression used in this study for determining the fraction of fuel which was oxidized was

$$\frac{\left(C_3H_8\right)_{in} - \frac{THC}{3}}{\left(C_3H_8\right)_{in}}$$

where $\left(C_{3}H_{8}\right)_{in}$ is the inlet volumetric concentration of propane and THC is the volumetric concentration of unburned hydrocarbons in the catalyst exhaust measured by the flame ionization detector. Because this analyzer measures total hydrocarbons on the basis of carbon-atom concentration, the indicated hydrocarbon concentration has to be divided by three to obtain the equivalent-propane concentration.

Hydrogen concentration was not measured in this study; therefore, any propane converted to hydrogen would appear to be fully oxidized in the data.

The measured exhaust carbon monoxide concentration for all catalysts and test conditions was between 10 and 20 ppm. This level is typical of background readings on the analyzer. The exhaust gas sample was not quenched after leaving the catalyst and reactions could have continued downstream. However, for temperatures much below 1100 K CO and hydrocarbon reactions are essentially frozen; therefore, it can be concluded that for most of the range of temperatures covered in this study, CO did not contribute to the inefficiency of oxidation, and the fraction

of fuel which was oxidized can be determined solely from the hydrocarbon measurements.

Figures 4(a) to (g) give the propane oxidation performance of each of the catalysts tested in alphabetical order by manufacturer. The catalyst bed temperature, which is the abscissa in these plots, differed from the exit temperature by less than 50 K in every test. The difference was usually about 30 K.

The results of the tests of both American Cyanamid and Chemetron pellets are shown in figure 4(a). From table I it can be seen that the American Cyanamid catalyst is a combination of platinum and palladium while the Chemetron catalysts have varying loadings of platinum. The G43 catalyst, with both platinum and nickel, gave the lowest oxidation of the five American Cyanamid and Chemetron pellets tested. Figure 4(a) shows that the other four pellets had some activity at temperatures as low as 325 K and a fairly constant oxidation of about 10 percent between 400 and 500 K. For higher temperatures with the Chemetron catalysts, the amount of fuel oxidized at a given temperature increased with the platinum loading. Although the American Cyanamid catalyst had a lower noble metal content than the Chemetron catalysts, it oxidized more propane between 550 and 850 K than any of the Chemetron catalysts. However, maximum conversion for it was only about 70 percent up to 900 K. The increase in propane oxidation for temperatures above 900 K may be due to the phenomenon of catalytically supported thermal reaction discussed in references 12 and 13.

For comparison with the catalytic oxidation of propane the thermal oxidation is also shown in figure 4. The thermal data were obtained using the same equipment and procedure as the catalyst tests except that a 5-centimeter length of cordierite monolith substrate occupied the usual position of the catalyst sample. Feed gas was 514 ppm propane in air at space velocities of 53 000 to 56 000 hour $^{-1}$ and a pressure of 2×10^5 newtons per square meter.

Between 1050 and 1150 K the measurements showed nearly complete thermal oxidation of propane. No significant CO concentration was measured. These measurements were not indicative of the results of thermal oxidation in a 5-centimeter length of cordierite monolith because the reactions could have occurred over the entire length of heated quartz tubing and could have continued into unheated downstream lengths of quartz tubing and gas sample line. When the temperature in the quartz tubing or sample line fell below about 1100 K, however, reactions should have been frozen. The thermal reaction curve serves to establish a temperature limit above which experimental data from catalyst tests is subject to error because of purely thermal reactions within the test apparatus. For this reason no catalyst test data are reported above 1050 K.

The thermal data indicate about 3.5 percent thermal oxidation at temperatures below 900 K; in terms of hydrocarbon analyzer measurements, the furnace inlet concentration was 514 ppm and the exhaust was 496 ppm. Because this difference is comparable with the measuring accuracy of the analyzer, an indicated oxidation of ± 3 percent can be considered effectively nil.

Results of tests of four Engelhard monoliths are given in figure 4(b). These catalysts are described in table II. None of the catalysts had any activity below about 450 K; however, the EVD 1412, a palladium catalyst, gave nearly 90 percent oxidation at 600 K.

W. R. Grace and Company catalysts were tested in both the pellet (fig. 4(c)) and monolith (fig. 4(d)) form. In addition, the Grace Davex 300, a palladium "minilith," shown in figure 3 was tested. The minilith had a higher noble metal loading than the Grace pellets (see table I) and oxidized more propane between 550 and 700 K. For higher temperatures, the other palladium catalyst, the Davex 168 M, showed superior activity. Possibly the open structure of the minilith resulted in the reactions at the higher temperatures being limited by diffusion of fuel and oxygen molecules to the catalyst surface (refs. 12 and 13). Within the accuracy of the data the results for the Davex 145 and 167 were the same.

The Grace noble metal monoliths (fig. 4(d)) were all tested with identical substrate configurations. The Davex 512A, 512B, and 517 gave similar results between 550 and 600 K. At temperatures above 600 K propane oxidation was more complete with the Davex 517 than with the 512A or 512B. At temperatures between 400 and 500 K, the Davex 512A and 512B had a plateau of about 10 percent conversion while the 517 decreased to zero. The Davex 516 is a base-metal-oxide catalyst; its oxidation curve fell to the right of the noble metal results. The rare-earth oxides, the Davex 519, 520, and 521 required relatively high temperatures before activity was apparent.

Two Matthey Bishop monoliths were tested. They gave nearly identical results and are plotted together in figure 4(e). Although activity was apparent even at 325 K, 90 percent oxidation required a bed temperature of over 950 K.

All the monolithic catalysts tested had substrate cell densities of between 30 and 45 cells per square centimeter with the exception of the Oxy-Catalyst samples which used a substrate with only 10 cells per square centimeter. Because the total substrate surface area of these catalysts is lower than for the other monoliths tested, the ability to oxidize propane might also be expected to be poorer. For temperatures below 600 K, however, the propane oxidation was among the best of the catalysts tested (see fig. 4(f)). But oxidation reached a peak of less than 70 percent at a temperature of 750 K and decreased with increasing temperatures up to 900 K. The catalytically-supported thermal oxidation discussed in references 12 and 13 is probably responsible for the increase in activity which

occurs at temperatures above 900 K. The decrease in activity between 750 and 900 K may result in part from a diffusion-limited reaction rate and an increasing gas velocity (therefore, a decreasing residence time) as bed temperature increases. It is likely that this unusual effect results from the large cell diameter of the substrate (0.32 cm). Because of the high activity displayed at low temperatures a smaller cell (on the order of 0.16 cm) might be expected to give performance similar to that of the Engelhard EVD 1412, Grace Davex 512A, Davex 512B, or Davex 517. It is interesting to note that none of the other monoliths tested displayed any diffusion-limiting effects.

Figure 4(g) shows the performance of two pellets and two monoliths made by Universal Oil Products. As with many of the other catalysts tested, activity is seen at very low temperatures with a plateau of about 10 percent oxidation until higher temperatures are reached. The B pellet performed nearly as well as any of the pellets tested up to about 750 K, then reached a plateau before oxidation increased with temperature for temperatures above 900 K. The results for this catalyst were very similar to those for the American Cyanamid catalyst. The UOP monoliths had lower noble metal loadings than some of the other monoliths tested (see table II). As a result, at high temperatures the fraction of fuel oxidized tends to be lower.

Effect of Space Velocity on Propane Oxidation

The effect of variations in space velocity on the propane oxidation is shown in figure 5. The space velocity was varied in two ways: by changing the mass flow rate and by changing the catalyst volume.

In figure 5(a) the effect of varying the mass flow rate on the performance of the Oxy-Catalyst catalyst 1 is given. For an increase in space velocity from about 45 000 to 60 000 hour⁻¹, the propane oxidation curve is shifted down about 10 percent for temperatures above 500 K.

When the tested volume of the Grace Davex 169 pellet catalyst was doubled from 13 cubic centimeters to 26 cubic centimeters, no change in oxidation was observed for the space velocities tested, as shown in figure 5(b).

These results show that the variation in space velocities at which the various catalysts were tested probably had little effect on the results.

Effect of Initial Propane Concentration on Catalyst Performance

All but four of the catalysts were tested with initial propane concentrations of about 500 ppm. Two of the Oxy-Catalyst catalysts as well as the Matthey

Bishop catalysts were tested with 280 ppm propane. Figure 4(f) suggests that within the experimental scatter of the data initial fuel concentration had no effect on the performance curve.

CONCLUDING REMARKS

These furnace tests of propane oxidation have indicated that the most effective catalysts for gas turbine combustor applications will probably be noble metals on monolithic substrates. Optimum cell density will be determined by several considerations including pressure drop, catalytic performance, and mechanical strength; only the second criterion was made a part of this study, however, and results showed that of the structures tested, those with 30 to 45 cells per square meter provided the best performance.

Most of the catalysts tested in this study were developed for use as automobile exhaust emission control reactors. None of the catalysts were specifically designed for the oxidation of propane or optimized for the conditions of these screening tests. Thus, the results should not be considered a judgment on the capability of any of these catalysts for other applications, fuels, or test conditions.

None of the catalysts was subjected to the high temperatures (1400 to 1600 K) required for catalytic combustion; as part of the catalyst selection process it will be necessary to determine which catalysts maintain high levels of activity after aging at 1400 to 1600 K.

Catalyst evaluation will also be required at the velocities and fuel-air ratios typical of gas turbine combustor operation. Some tests have already been made in a 12-centimeter diameter combustion test rig using several Grace and Oxy-Catalyst catalysts (ref. 11).

The tests described here were only a first step in a long process of catalyst selection and combustor design which will be necessary before practical catalytic combustors can be demonstrated.

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TABLE I. - CATALYST PELLETS TESTED

Manufacturer	Designation	Active component(s)	Loading ^a	Substrate manufacturer	Substrate composition	Pellet shape	Pellet size, cm
American Cyanamid	Aero Ban	Pt Pd	260 g/m ³ 104 g/m ³	American Cyanamid	Alumina	Cylinder	0.32 diam. by 0.45 long
Chemetron Corp.	Girdler T309	Pt	0.1%	Chemetron Corp.	Alumina I	Cylinder	i i
	Girdler T309B	Pt	0.5%			Cylinder	by 0, 32 long 0, 32 diam, by 0, 32 long
	Girdler T1710	Pt	0.15%			Sphere	0.32 diam.
	Girdler G43	Pt	0.1%			Cylinder	0.64 diam.
·		Ni	3%	, Y	٧		by 0.64 long
							·
W. R. Grace and Company	Davex 145	Pd	NA	W. R. Grace	Alumina	Sphere	0.35 diam.
	Davex 167	Pd-promoted base metal	100 g/m ³ Pd			Cylinder	0.3 diam. by 0.44 long
	Davex 168 M	Pd	360 g/m^3			Cylinder	0.34 diam. by 0.55 long
	Davex 169	Pt/Pd: 5/2	360 g/m^3	·	\	Sphere	0.4 diam.
	Davex 300	Pd	0.4%	*	Mullite	Cylinder	0.57 diam.
				·			by 0.63 long
Universal Oil Products	A	Pt	585 g/m ³	UOP	Alumina	Sphere	0.4 diam.
	В	Pt	135 g/m^3	UOP	Alumina	Sphere	0./4 diam.
		Pd	525 g/m^3				

^aCatalyst loading is the weight of catalyst applied to the substrate. It is expressed either as a percent of total substrate weight or as weight per unit substrate volume. Most pellets have a bulk density of about 650 kg/m 3 ; thus, 1% loading is equivalent to about 6500 g/m 3 .

TABLE II. - MONOLITH CATALYSTS TESTED

Manufacturer	Designation	Active component(s)	Loading ^a	Substrate manufacturer	Substrate composition	Cell density, cells/cm ²	Cell shape
Engelhard Industries	EVD 1057 EVD 1128 EVD 1337 EVD 1412	Pt, Pd Pt Pt Pd	NA NA NA NA	Corning	Cordierite	35	Corrugated
W. R. Grace and Company	Davex 512A 512B 516 517 519 520 521	$\begin{array}{c} \text{Pt, Pd} \\ \text{Pt, Pd} \\ \text{CoO} \\ \text{Pd} \\ \text{CeO}_2 \\ \text{La}_2\text{O}_3 \\ \text{CeO}_2, \text{La}_2\text{O}_3 \end{array}$	NA NA NA 0.3% NA NA	W. R. Grace	Cordierite	45	Square
Matthey Bishop	AEW23/20G/40M AEW23/26G/40M	Pt Pt/Pd: 2/1	1700 g/m ³ 1700 g/m ³	Corning Corning	Cordierite Cordierite	31 31	Square Square
Oxy-Catalyst	1 2 3	Pd/Pt: 2/1 Pd/Pt: 2/1 Pd/Pt: 2/1	1500 g/m ³ 1800 g/m ³ 1600 g/m ³	General Refractories Norton Norton	Cordierite Silicon carbide Silicon carbide	10 10 10	Round Round Round
Universal Oil Products	C	Pt Pd	879 379	Corning	NA	36 36	Triangle
	D	Pt	629	Corning	NA	36	Triangle

^aCatalyst loading is the weight of catalyst applied to the substrate. It is expressed either as a percent of total substrate weight or as weight per unit substrate volume. Most monoliths have a bulk density on the order of 250 kg/m^3 ; thus 1% loading is equivalent to about 2500 g/m^3 .

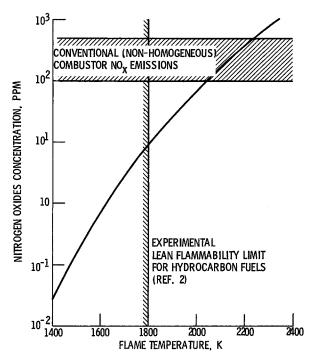
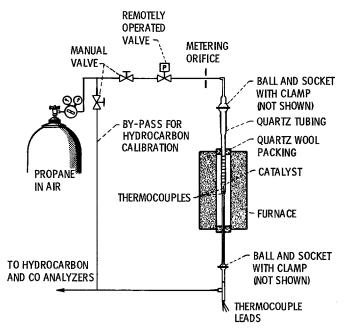


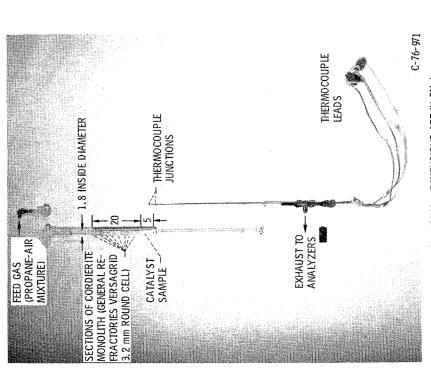
Figure 1. - Effect of flame temperature on nitrogen oxides emissions. Well-stirred reactor (ref. 1) with residence time of 2x10⁻³ sec; pressure, 5.5x10⁵ newtons per square meter; inlet mixture temperature, 800 K.



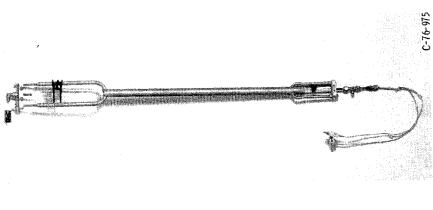
(a) TEST SYSTEM SCHEMATIC (SUPPORTING FRAME FOR QUARTZ TUBING NOT SHOWN)

Figure 2. - Test apparatus.

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(b) CATALYST-TEST TUBING. (DIMENSIONS ARE IN CM.) Figure 2. - Test apparatus.



(c) TJBING INSTALLED IN SUPPORTING FRAME Figure 2. - Test apparatus.

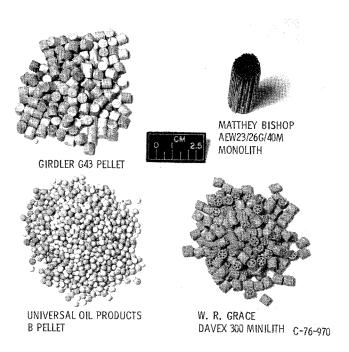


Figure 3. - Typical catalyst samples.

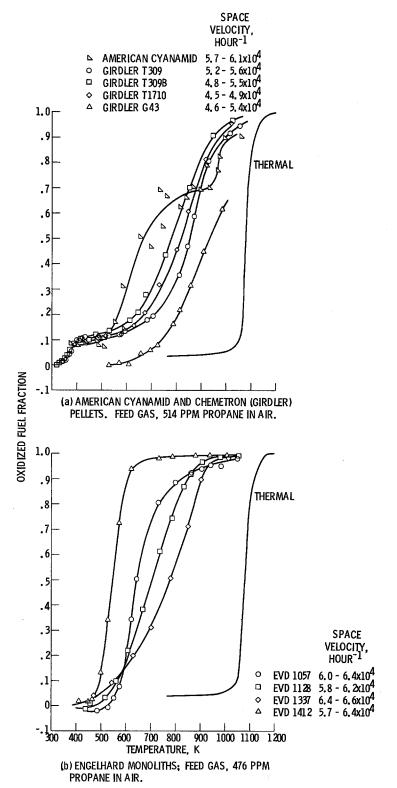


Figure 4. - Catalyst performance; pressure, 2x10⁵ newtons per square meter.

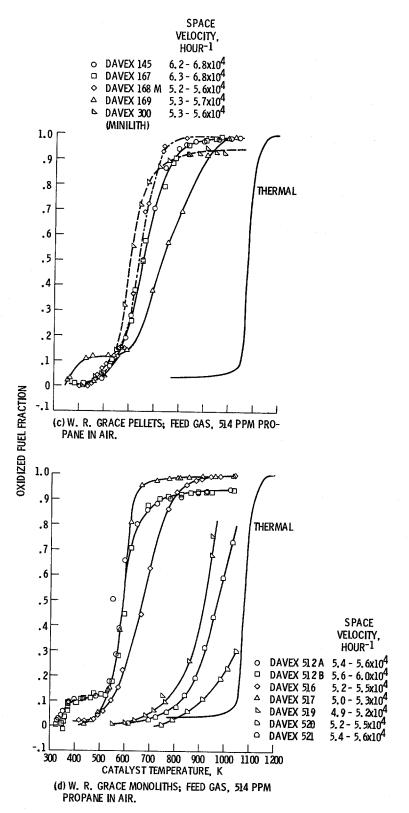


Figure 4. - Continued.

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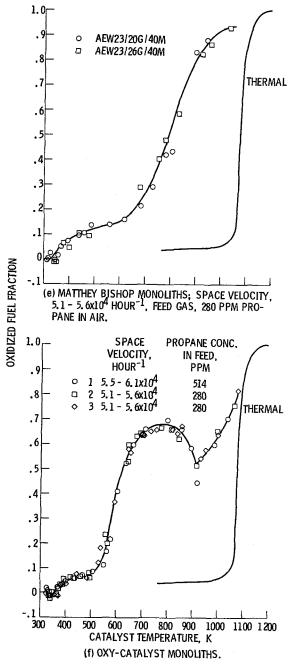


Figure 4. - Continued.

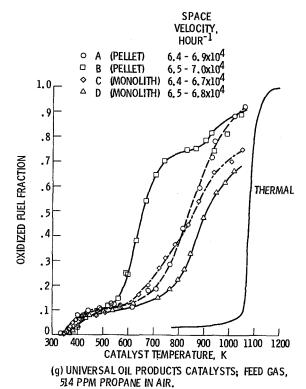
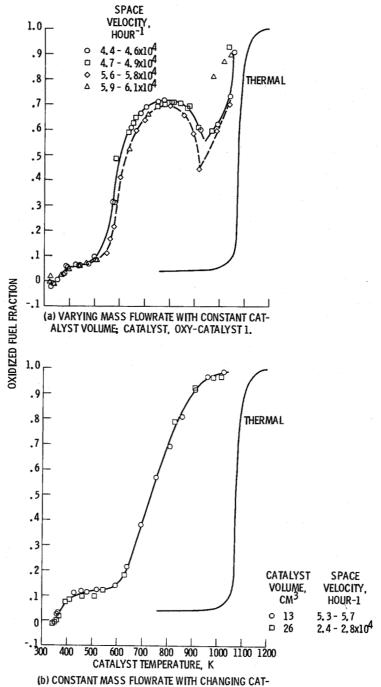


Figure 4. - Concluded.



ALYST VOLUME; CATALYST, GRACE DAVEX 169.

Figure 5. - Effect of space velocity on hydrocarbon oxidation. Pressure, $2x10^5\,N/m^2$; feed gas, 514 ppm propane in air.